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~~Structure of Atomically Clean Surfaces of Refractory Metals~~  
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STRUCTURE OF ATOMICALLY CLEAN SURFACES OF REFRACTORY METALS

by

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The structure of a surface cut parallel to a (110) plane in a tantalum single crystal has been studied as a function of heat treatment and ion bombardment for a limited range of conditions. Prior to mounting in the vacuum system, the crystal was

- 1) mechanically polished until a mirror surface was obtained,
- 2) chemically etched with a 5:2:2 sulfuric acid, conc. nitric acid, 48% hydrofluoric acid mixture,
- 3) again mechanically polished to a mirror surface,
- 4) electropolished for 10 minutes in a 1:4:4, 48% hydrofluoric acid, conc. hydrofluoric acid, distilled water solution. The cathode was carbon, and the dc voltage applied to the cell was 25 volts with a resulting current density of 0.05 - 0.2 amp/cm<sup>2</sup>.

The crystal has been heated in the temperature range 900-1100°C for 320 hrs and ion bombarded for a total time of 64 minutes. The programming of the heat treatment and ion bombardment together with the data obtained subsequently were interpreted with the following conclusions:

- 1) ion bombardment and anneal does not measurably change the structure of the surface,
- 2) heat treatment does change the structure of the surface,

- 3) the changes in surface structure are not reversible with temperature.

The evidence for the above statements will be discussed qualitatively. The crystal was alternately outgassed at  $900^{\circ}\text{C}$  and ion bombarded with 500 ev argon ions, the total heating time was 65 hrs and the bombarding time 23 minutes. The ion bombardments were followed by low temperature annealing at dull red heat. The diffraction pattern observed was characteristic of a surface composed of (130) facets. It appears probable that the electropolishing treatment was responsible for the production of (130) facets on the crystal surface. The crystal was then heated for an additional 240 hrs at  $900^{\circ}\text{C}$  with periodic ion bombardment totaling 12 minutes (using 500 ev ions and low temperature anneal as before). The observed diffraction pattern was again characteristic of (130) facets and the intensities of the diffraction beams were essentially the same as before. Thus, a 50% increase in total bombarding time caused no observable change in surface structure. Further, it was found that slow cooling of the crystal from  $900^{\circ}\text{C}$  to room temperature, as compared to radiation quenching the crystal from  $900^{\circ}\text{C}$ , resulted in the same diffraction pattern. This indicates that the facets present at  $900^{\circ}\text{C}$  were stable at all temperatures down to room temperature.

Heating the crystal to  $975^{\circ}\text{C}$ , radiation quenching, ion bombarding, and annealing at low temperature caused a slight

change in surface structure. That is, there appeared some diffraction beams characteristic of (110) facets, in addition to the beams characteristic of (130) facets. Repeating the above experiment at 1000°C further increased the strength and number of the (110) diffraction beams. Again, a radiation quenching and slow cooling experiment at 1000°C gave the same diffraction pattern. These observations confirm the irreversibility of the surface change since the pattern characteristic of a 900°C heat treatment was not obtained when the crystal was slowly cooled through the 900°C temperature range. Additional one-hour heatings of the crystal at 1050°C and 1100°C produced further increases in the intensities of the (110) diffraction beams.

To confirm that the structure changes were due to the heat treatment and not to the ion bombardment, the following experiments were performed. The crystal was ion bombarded for 10 minutes with 500 ev ions, annealed for 1 hr at low temperature and the diffraction pattern observed. The crystal was then ion bombarded with 1000 ev ions and annealed for 1 hr at the same low temperature as before. The diffraction pattern was the same in both cases and the same as that observed after the 1100°C heat treatment, thus indicating no effect of ion bombardment on surface structure. The only difference was that the beams associated with the ion bombarded surfaces were

not as intense as those associated with the surface after 1100°C heat treatment.

The effect of bulk contamination diffusing to the surface of the crystal has been observed. As mentioned above, diffraction beams characteristic of a surface composed of (130) facets were observed in the early stages of the heat treatment. These beams were observed in the (001) azimuth, while in the other major azimuths,\* the (1 $\bar{1}$ 1) and the (1 $\bar{1}$ 0), no ordered beams were observed. On the other hand, as the heat treatment proceeded, beams characteristic of diffraction from (110) facets were observed in all three major azimuths. Furthermore, in the (1 $\bar{1}$ 1) and (1 $\bar{1}$ 0) azimuths, the diffraction beams followed first-order surface grating curves, indicating diffraction from a surface with essentially the same atomic spacing as that of the bulk (in these azimuths). At the same time, diffraction beams were also observed, in these azimuths, which followed  $\frac{1}{2}$ -order surface grating lines to a first approximation. In the case of metals,  $\frac{1}{2}$ -order beams are usually characteristic of gas adsorption. In this case the beams did not follow the theoretical  $\frac{1}{2}$ -order curves exactly and low temperature heating of the crystal ( $\sim 900^\circ\text{C}$ ) failed to remove them. Further work will be required to determine the source of these beams. Finally, the (110) beams observed in the (001) azimuth did not follow the first-order surface grating curves or the  $\frac{1}{2}$ -order, but could be interpreted as high order beams from a (110) surface with a large lattice constant.

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\*An azimuth is defined as the crystallographic plane containing both the incident and diffracted electron beams.